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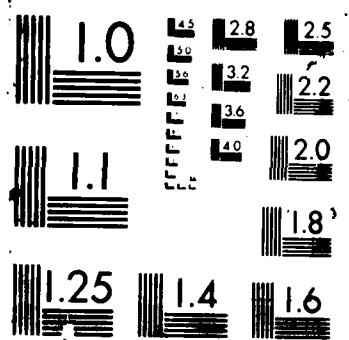
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Development of facilities for studies of high temperature decomposition of solid propellant ingredients are described, particularly as they pertain to use of the subject DoD Equipment Grant. Funding from the Grant was used for laboratory support equipment and peripheral equipment on existing experiments, but primarily for purchase of a 1200 watt CO<sub>2</sub> laser and related beam optics and support equipment. This apparatus will permit heating of ingredient samples at surface heat fluxes common in propellant combustion, involving temperature rise rates of 10<sup>5</sup> °C/sec at the surface. Combustion research indicates that decomposition mechanisms are usually different in such high heating rate situations, giving considerable urgency to development of high rate experiments.

Several other planned uses for the laser facility are also noted.

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**SCIENTIFIC AND FINAL REPORT**

**COMBUSTION DYNAMICS OF SOLID PROPELLANTS**

By

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Prepared for

AIR FORCE OFFICE OF SCIENTIFIC RESEARCH  
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## BACKGROUND

Georgia Tech has maintained research studies in the School of Aerospace Engineering on combustion of rocket propellants and energetic systems since 1967. Contracted research has been sponsored by Navy, Air Force, Army, NASA, and industrial funding as well as University funding. A laboratory in the School of Aerospace Engineering has been equipped for this research, and approximately 35 graduate degrees have been awarded to students conducting research in this area. This specialized research is carried out in the context of a broader research and teaching program in aerospace propulsion and related scientific disciplines involving some 10 teaching faculty and 12 engineering staff members.

Combustion of solid propellants and solid ramjet fuels (and filled polymers in general) involves decomposition of the solid ingredients to vapor products, which react exothermally in the gas phase to provide all or part of the heat that sustains the surface pyrolysis. The pyrolysis rates of the solids that are required for practical applications require temperatures around 600-700° C. In the combustion environment, these temperatures are reached very quickly in a thermal wave that propagates into the solid ahead of the "burning" surface. Only in the presence of such rapid heating (e.g.,  $10^5$  °C/sec) is it possible to have decomposition at such high temperatures. At lower heating rates, the sample is gone before such temperatures are reached.

The propellant combustion wave is a very difficult environment in which to study ingredient decomposition, so many experiments have been contrived that are more suitable for conduct of measurements and analytical description of processes. Such methods include self-deflagration of ingredients that can sustain such behavior (e.g., ammonium perchlorate), combustion of geometrically or chemically simplified systems (e.g., "sandwich" burning and fuel combustion in oxidizing gas environments), and decomposition during externally controlled heating. All of these methods are in use together in the studies of propellant combustion at Georgia Tech. Each method has its own merits and weaknesses, but collectively they offer the prospect of definitive understanding of propellant combustion. The merit of the simpler experiments derives from their susceptibility to more definitive control of variables, measurement of processes, and interpretation of results. However, in the present context, the value of results depends on their relevance to propellant combustion. Relevance can easily be unintentionally sacrificed in the pursuit of "measurability" and

"understandability". It is increasingly evident that this has happened in the area of controlled decomposition experiments, and the central objective of the facility development under the present contract has been to provide ingredient decomposition experiments that provide testing conditions that better simulate the combustion zone environment. The primary thrust to date is to develop experimental facilities that will permit rapid controlled heating of test samples to temperatures in the 500-1000° C range, and to measure the rates of decomposition and the composition of decomposition products in the absence of complicating combustion reactions. Current plans call for experiments in inert gas at atmospheric pressure. Later, work will be extended to lower and higher pressure. These goals were set as a result of findings in past and current DoD contracts (Refs. 1-4), and led to the proposal on which the present contract was based. In the following, a description will be made of experimental methods in use in the ongoing research, and the use of the DoD Equipment Funds to equip test facilities for that research. Some other potential applications of the facilities will also be listed to suggest lines of future research. A subsequent section of the report will describe what was purchased under the present contract, and the present and projected utilization.

#### DECOMPOSITION EXPERIMENTS

The objective of any high temperature decomposition experiment is to produce a controlled heating of a sample in a chosen environment, and to observe what happens to the sample. The experiments can be made relatively simple by "suitable" choice of the heating and environment, and by limiting the tests to simple materials. For example, if the sample is heated slowly enough, it will be easy to measure its temperature, and the temperature will be spatially uniform so that the temperature time history of all of the sample will be the same. Temperatures will be limited to relatively low values that can be reached before the sample is vaporized. When high temperature decomposition is sought, samples must be heated rapidly. It is not generally possible to maintain spatially uniform temperatures in a sample when it is heated rapidly, and it is difficult to determine the space-time-temperature history. The present facilities have struck three levels of compromise in choice of facilities:

1. Conventional experiments in which samples are heated by electrical heaters at rates of up to  $1^{\circ}\text{C/sec}$  (hot stage microscope (optical and scanning electron types), differential thermal analyzer, thermogravimetric analyzer).
2. A relatively high heating rate thermogravimetric analyzer (HHRTGA), designed and built in this lab (Ref. 2), that can use radio frequency inductive heating or laser heating and make time-continuous mass and temperature measurements for heating rates to  $10^2\text{ }^{\circ}\text{C/sec}$  (spatially uniform sample temperature is achieved by using very thin samples).
3. A very high heating rate decomposition facility that utilizes a 1200 watt  $\text{CO}_2$  laser to produce heating rates to  $10^5\text{ }^{\circ}\text{C/sec}$  (higher rates with a focused beam). By this means, test samples will be heated by passage of a thermal wave similar to that during combustion, and pyrolysis rate will be measured by surface regression rate and/or sample weight change. The heating history of individual volume elements will be determined from measured surface temperature and surface regression rate combined with calculation of corresponding temperature-time histories (which will be verified by thermocouples embedded in the samples).

The three types of facilities noted above are useful for developing different kinds of information about how samples decompose. The conventional systems are relatively easy to operate, and useful for determination of properties of samples such as temperatures of crystal phase change and/or melting, onset of vaporization, and estimates of energies of phase changes and apparent activation energies of decomposition reactions. Since most published literature is based on these low rate experiments, it was deemed advisable to upgrade the capability in this laboratory so that results of high rate tests could be compared with the "conventional data base" (to determine the conditions under which high heating rates and temperatures lead to different decomposition mechanisms). The upgrading included purchase of a Perkin and Elmer Thermal Analysis Lab Model 7/4; purchase of accessories for an available Leitz optical microscope and its camera attachments for use as a hot stage microscope. A video recorder was purchased for use with the available scanning electron microscope, to record sample behavior during heating (in vacuum).

The high heating rate thermoanalyzer was developed with ONR Contracts N00014-79-C-0764 and N00014-85-K-0803 and is described in detail in Refs. 1 and 2. The test sample is deposited as a thin film on a ferroelectric sample holder that is heated by RF induction. Temperature is measured by a thermocouple attached to the sample holder. RF energy is provided by a Fischer 0310 Curie Point pyrolizer unit (The sample was also heated with the heater unit from the Perkin and Elmer system for comparison). The heater element is mounted on the free end of a vibrating quartz tube, and the change in sample mass is measured by the change in frequency of the vibration. The system was used to determine the effective activation energy of HTPB and PBAN binders at high and low heating rates. The indicated activation energy of HTPB was ~ 80,000 cal/mole at a heating rate of 100° C/sec, as compared to a value of ~ 18,000 at conventional heating rates, e.g., 1° C/sec. PBAN binder showed the same low value of activation energies as HTPB, but at both heating rates. This result indicates the importance of obtaining decomposition behavior at high heating rate. The low activation energies appear to reflect rate control by evaporation, while the high activation energy appears to reflect rate control by chemical bond breaking. Under the present contract, the primary support of this HHRTGA facility was the purchase of three amplifiers for use in the data acquisition systems.

The CO<sub>2</sub> laser pyrolysis facility is intended for a variety of ignition, pyrolysis, and combustion experiments (Ref. 5). The immediate applications are to ONR Contract N00014-85-K-0803 (ingredient pyrolysis) and ARO Contract DAAG 29-85-K-0125 (thermite combustion). This facility is still under construction; the laser is operational and in use with improvised experimental setups, while the optical train, control apparatus, test chambers, and measurement systems are under construction. This facility has been the primary cost item under the present Equipment Grant. A decision was made in concert with the AFOSR Technical Monitor to acquire a 1200 watt laser instead of the proposed 500 watt unit, a decision based on potential added usefulness and advances in available commercial lasers. The manufacturer also provided a \$25,900 grant toward the purchase. This facility has already made it possible to obtain controlled ignition, laser-assisted burning, and interrupted burning of metal-metal oxide samples for the above-noted ARO contract. When completed, the facility will provide a uniform beam for the pyrolysis of polymer (fuel) samples at temperature rise rates of 10<sup>5</sup> °C/sec. A follow-on DoD equipment grant is concerned with instrumentation for measurement of sample behavior during the heating-pyrolysis

event (time resolved temperature and composition fields during high rate pyrolysis and oscillatory pyrolysis).

## CURRENT AND PLANNED RESEARCH

The high temperature decomposition facility provides the means for a wide range of research, not only in propulsion applications but in all high temperature applications and problems from fire research to determination of material properties, to metallurgy, to material failure. The propulsion-related problems currently planned or in progress are:

1. Decomposition of polymeric materials, such as propellant binders and ramjet fuels (in progress),
2. Combustion of "thermite" materials (gasless combustion of metal-metal oxide and metal-metal systems) (in progress),
3. High temperature behavior of particulate burning rate catalysts, such as  $\text{Fe}_2\text{O}_3$ , and mechanisms of catalysis (in progress),
4. Sintering and agglomeration processes in metal powders used as fuels in propulsion,
5. Oscillatory combustion using oscillating laser beam as a controlled perturbation source,
6. Decomposition of solid oxidizers: topochemistry of crystal decomposition, presence of melts at high temperature, composition of vapor products,
7. Laser assisted combustion to permit combustion at low pressure with extended combustion zone (making space-resolved combustion zone diagnostics practical),
8. Ignition mechanisms, ignition energy requirements.

## EQUIPMENT PURCHASED AND INSTALLED

A listing is provided below of equipment purchases and installation costs charged to this Grant and Georgia Tech matching funds. The items are grouped according to the particular experimental facilities noted in the text.

### CO<sub>2</sub> Laser Pyrolysis Facility

Penn Research Corporation, 1200 Watt Laser	122,500
Laser Beam Benders and Lens	12,661
Optical Bench	5,314
Gas Regulators	1,105
Exhaust System Blower	1,357
Water Chiller	2,500
Installation costs (moving, electrical, water hook up)	1,132

### Thermal Analysis Lab

Perkin and Elmer DTA, TGA (System 7/4)	29,395
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### Optical Hot Stage Microscope

Leitz Mirror Housing	1,760
Motor Drive and Film Magazine for Nikon Camera	1,137
Lens for Nikon Camera	170
Lens for Lo Cam Camera	150

### Scanning Electron Hot Stage Microscope

Video Recorder	300
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### High Heating Rate Thermogravimetric Analyzer

3 Neff Amplifiers	2,471
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Test Sample Preparation Equipment

Mettler Balance	1,895
Milli Balance (Cahn DTL 7500)	2,033
Sonic Sifter	6,126
Micro Sieves	1,092
High Temperature Bath	386

General Lab Equipment

Lockable Storage Cabinets (five)	1,175
Motion Picture Analyzer (16 mm stop motion projector)	2,465
Monochromatic Light (sodium)	797
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	\$ 197,921

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5. Price, E. W., "Status Report on DoD/AFOSR Grant AFOSR-84-0183, Amend. A," Georgia Institute of Technology, Atlanta, GA, December 1985.

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